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Peter Blanchette
Stephen F Austin State University

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ANGELINA COUNTY, TEXAS

APPROVED:

Dr. Kenneth G. Watterston,
Thesis Director

Dr. James E. Howard,
Committee Member

Dr. David L. Kulhavy,
Committee Member

Glen T. Clayton, Dean of the
Graduate School

SULFATE LEACHING FROM SULPHUR BINDING AGENTS IN EXPERIMENTAL ASPHALT PAVEMENT, ANGELINA COUNTY, TEXAS

by

PETER LYNN BLANCHETTE, B.S.F.

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INTRODUCTION

At the onset of the 1980's, the United States is experiencing a rapidly increasing demand for energy resources. This demand has increased our search for alternative energy sources, methods of energy conservation and substitutes for petroleum products. The Arab oil embargo of the early 1970's and the price fixing action of the Organization of Petroleum Exporting Countries (OPEC) also has intensified our nation's need for these energy alternatives and conservation of our present nonrenewable resources.

It is recognized that coal is the major energy alternative in the United States in sufficient quantities and low enough in price to fulfill our imminent needs (Tomimatsu and Johnson, 1976). The major disadvantage of coal is that it has more airborne pollutants than gas and oil. The entrapment and recycling of hydrocarbons, carbons and sulphur byproducts from the use of fossil fuels is essential. Many corporations have started research and use of sulphur byproducts in materials such as concrete, foam, cardboard, batteries, ceramics, building materials and asphalt (Meyers, 1977).

Since the early 1970's field trials using sulphur-asphalt have been conducted. Shell Canada Ltd. in 1973, used sulphur-asphalt (trademarked "Thermopave") on road trials in Canada (Sulphur Institute, 1973a). In 1973, the Societe Nationale des Petroles d' Aquitaines (SNPA) of France established a road test at Lacq in southwestern France (Sulphur Institute, 1973b). Sulphur-asphalt was also used to

patch cracks at McCarran International Airport in Las Vegas, Nevada, after conventional asphalt failed (Sulphur Institute, 1976). In 1975, SNPA established a road test in Angelina County, near Lufkin, Texas, on U.S. Highway 69 in cooperation with the Federal Highway and Public Transportation (Sulphur Institute, 1975).

Thus far the road trial test dealing with the physical properties of sulphur-asphalt has shown it to be superior in strength, durability and water resistance than conventional asphalt. However, the environmental effects of sulphur-asphalt binders have not been examined.

The objectives of this study were to evaluate the extent of sulfate leaching from the sulphur-asphalt and its possible interrelationship with other soil cations and anions. These data will provide information for subsequent use of sulphur-asphalt binders in similar temperate regions.

LITERATURE REVIEW

Over a century ago sulphur was widely used in industry and chemistry labs. Sulphur discussions also filled about a third of every chemistry text. During the following decades sulphur research and use declined in chemistry labs and finally disappeared from discussions in college chemistry textbooks (Meyers, 1977). The decline in sulphur research and use paralleled the increase in petroleum technology. Fossil fuels became the major energy sources for mass production of manufactured goods. Sulphur research virtually halted until the early 1960's, when public concern for polution abatement increased (Meyers, 1977). These concerns fueled a renewed interest in the reestablishment of sulphur research. Further research was needed to document the behavior of soil sulfate mobility and its influence on the surrounding environment.

To discuss possible effects arising from the leaching of sulfate from sulphur-asphalt, it is imperative to recognize the sources and variables that influence sulfate mobility in the soil. Apparently, sulphur chemistry follows a biological cycle similar to nitrogen. In humid regions the major source of sulfate, the form available to plants, is released in the soil as a result of microbial activity (Starkey, 1950).

The absorption and desorption of the sulfate anion by soils is influenced mainly by the following soil variables: concentrations of calcium, iron, aluminum and phosphates which predominate under vary-

ing and specific pH conditions (Brady, 1974). Ensminger (1954) noticed that as liming increased pH, sulfate retention decreased. This may be due to precipitation of calcium sulfate leaving the anion exchange available to the increasing amount of hydroxyl ions in the soil solution (Mehlich, 1964). This agrees with earlier work of Chao, Harward and Fang (1963) showing that clay exchange complexes dominated by mono- and di-valent cations are less retentive of sulfates than soils dominated by iron and aluminum sesquioxides. In acid conditions where iron and aluminum sesquioxides dominate the clay exchange complex, sulfate retention is greater than when the clay is dominated with other cations (Chao et al., 1962b). This is probably due to the development of positive charges on the hydrous iron and aluminum oxides enabling them to absorb anions such as sulfates at very low pH's. Following the same concept, Chang and Thomas (1963) were interested in the effects released hydroxyl ions would have on pH when replaced by sulfate ions. Their results showed that such a reaction increased the hydrolysis of aluminum, by releasing hydrogen ions which would neutralize excess hydroxyl ions in solution, over time.

Finally, as phosphates increase in the soil, the amount of sulfate absorbed decreased (Ensminger 1954; Kamprath et al., 1956; Berg and Thomas 1959; and Chao et al., 1962a). The loss of sulfate due to the phosphate addition is a result of competition in the anion exchange mechanism (Chao et al., 1962a).

Examination of these variables is a necessary point of research concerning sulfate leaching from the soil profile. Coarse textured

soils, abundant precipitation, and small quantities of allophanes are conducive to high sulfate leaching. McKell and Williams (1960) conducted a lysimeter study, using S^{35} contained in gypsum, on a Vista sandy loam (coarse-loamy, mixed, thermic member of Typic Xerocrepts) in California. They fertilized at rates of 100 and 300 lbs. of gypsum per acre and were able to account for 77.0% and 77.9% respectively, of the sulphur applied after a season of heavy rainfall. Hague and Walmsley (1974) also used S^{35} in a sandy loam and a clay from the Carribbean. Under the various water regimes applied, downward movement of sulfate was higher in the clay than in the sandy loam. The probable causes for these results were the clay's 6.7 pH and high organic matter content of the sandy loam. Thus, the behavior of each soil toward sulfate leaching results from several variables which are related to the soil's chemical and physical properties.

METHODS

Site Description

From September 1 through September 17, 1975, the Federal Highway Administration, the Texas State Department of Highways and Public Transportation, the Sulphur Institute and the Societe Nationales des Petroles d'Aquitaines, in cooperation with five other companies, constructed a test section for field evaluation of sulphur in asphalt binders on U.S. Highway 69, approximately twelve miles northwest of Lufkin, Texas. The test section is located on two newly constructed westerly traffic lanes between engineer stations 167+00 and 203+50. It is 3,650 feet long and two lanes wide, containing seven treatments and one control section (Figure 1). Soil samples were collected on both sides of the highway at station 202+00. Soil samples were also collected on both sides of the highway adjacent to three of the treatment sections at stations 196+00, 190+00 and 174+00 (Figure 1). The areas sampled alongside the road at these stations were approximately in the middle of each treatment section. Hereafter, the areas sampled will be referred to by the station number adjacent to where they were sampled. Surface and subsurface samples were also taken in the drainage ditches beside the test sections on the east and west sides. These ditches drain into a creek near station 188+50, where another sample was taken. Using these samples the degree of sulfate leaching into the creek can be evaluated.

The areas sampled are situated in gently rolling terrain on dis-

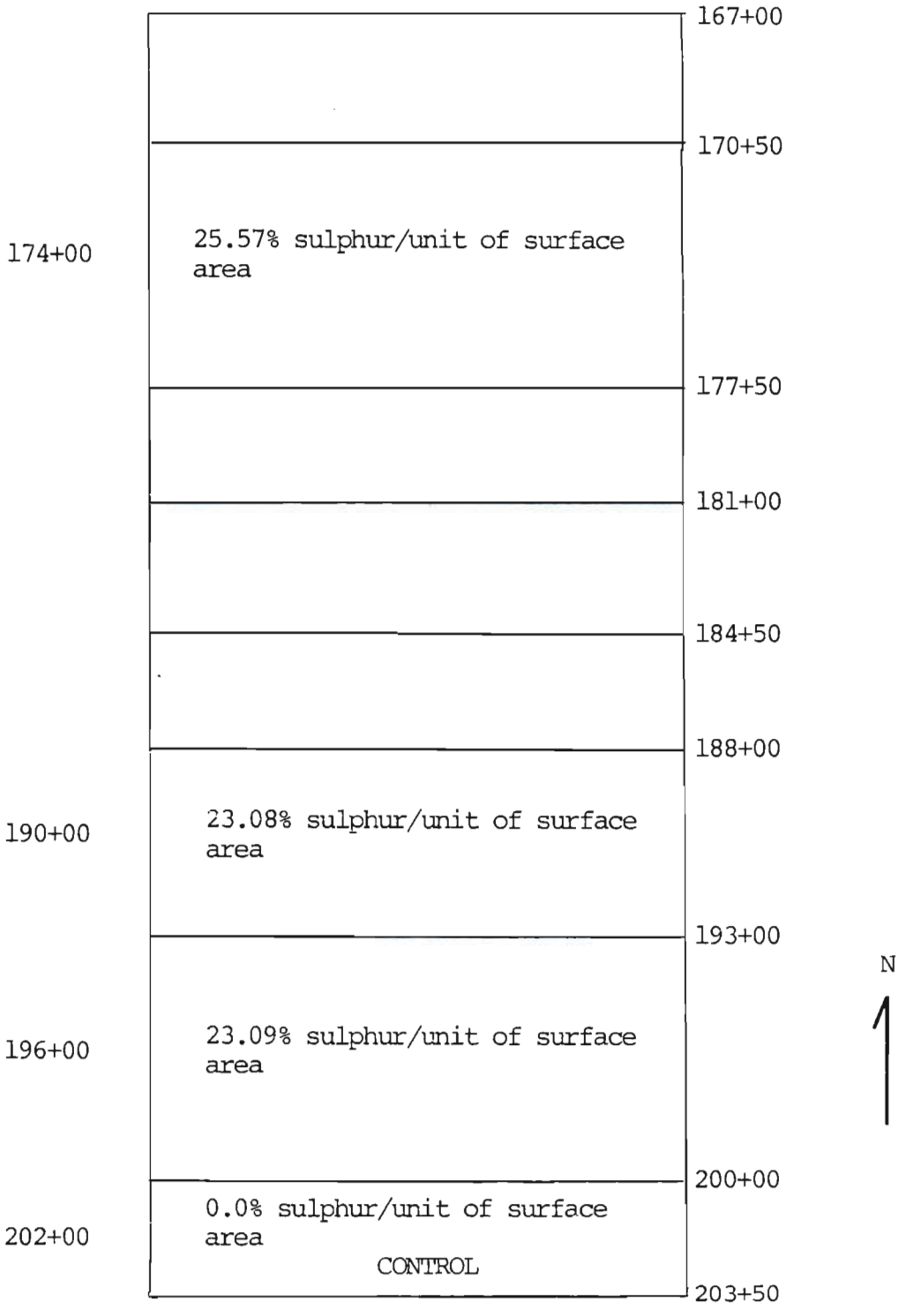

Figure 1. Test section with treatments delineated by engineer stations and interim stations adjacent to where soils were sampled.

turbed soils where a cut and fill operation lowered the northern half and raised the southern half of the test section. Although disturbed, these soils support an abundance of grass and forb species. Texturally, the soils are predominately sandy clay loams, clay loams and clays.

Construction and Materials Used

In preparing the site, two types of mineral aggregates were used. A fine graded sand, located in a heavily wooded area off Highway 103 about eight miles west of Lufkin, was used as a single aggregate in the preparation of a hot sand asphalt. The second type was a blend of 65 percent weight of crushed and screened gravel dredged from the Colorado River and 35 percent weight of the fine graded sand mixed to prepare a type "D" asphalt concrete. The hot sand and type "D" base materials were bonded with conventional asphalt containing no sulphur in control section 202. In all the treatment sections, 174, 190 and 196, a sulphur-asphalt mixture of 30 percent/70 percent by weight respectively was used as a binder. Information on what binder type the base materials were mixed with and where it was placed is shown in Table 1.

The test section was constructed in five layers. The first layer was a two inch hot sand asphalt "working table" throughout the test section. Layers two through five were paved using varying thicknesses and widths (Table 2). The lengths of the control 202 and treatments 196, 190 and 174 are 350 feet, 700 feet, 500 feet and 700 feet respectively. Each treatment contained varying percentages of sulphur-asphalt binder as shown in Table 3. Therefore, using Tables 1, 2 and 3, treatment lengths and the weight per one inch square yard of the type

Table 1. The type of pavement mixture used in each treatment, for each layer.

Treatment	Layers			
	2	3	4	5
174	SA-"D"	SA-"D"	SA-"D"	SA-"D"
190	SA-HS	SA-HS	SA-HS	SA-HS
196	SA-HS	SA-HS	SA-"D"	SA-"D"
202(control)	AC-HS	AC-HS	AC-"D"	AC-"D"

Nomenclature:

SA-"D" - Sulphur-in-asphalt binder in Type "D" asphalt concrete.

SA-HS - Sulphur-in-asphalt binder in Hot-Sand paving mixture.

AC-HS - Asphalt binder in Hot-Sand paving mixture.

AC-"D" - Asphalt binder in Type "D" asphalt concrete.

Table 2. The width and thickness of each layer in the test section.

Layers	Width in Feet	Thickness in Inches
5	28.00	1.50
4	28.75	1.50
3	29.50	2.00
2	30.00	2.00

Table 3. The percent binder in the asphalt of each layer as constructed and average percent binder for each treatment.

Percent Binder

Treatment	Layer				x % Binder
174	5.04	5.30	5.95	5.95	5.56
190	7.46	7.46	7.46	7.46	7.46
196	6.30	6.30	5.93	5.93	6.12
202(control)	5.67	5.67	5.04	5.04	5.36

"D" or hot sand asphalt, the tonnage of sulphur used per treatment can be determined. The type "D" and hot sand weigh 110 lbs. and 90 lbs., respectively, per one inch square yard.

Tonnages were determined as follows:

$$A = \frac{\frac{LW}{9} \cdot LT \cdot PYD}{2000 \text{ lbs.}},$$

where:

A = tons/treatment layer
LW = area of treatment (sq. ft.)
LT = layer thickness (in.)
PYD = lbs. asphalt/one inch square yard

This was done for layers two through five and summed for the total tonnage of asphalt per treatment (Table 4). Next, a ratio was set up to determine the tonnage of binder used per layer in each treatment from Tables 3 and 4 (Table 5). By multiplying 0.30 (the percentage of sulphur in the binder) by the tonnage of binder per layer in Table 5, the total amount of sulphur used per treatment was attained (Table 6).

Field Sampling

The soils were sampled at three locations perpendicular to each side of each treatment and control section of the pavement one, three and ten feet from the pavement (Figure 2). At each sampling location, a sample was taken at 0-3, 3-12 and 12-24 inch depths. Actual sampling locations within the area of each treatment were chosen adjacent to an engineers station nearest the middle of the test section. At the time of the first sampling, February 1976, all sample locations were surveyed and permanently marked by a Texas Highway Department

Table 4. Tons of asphalt in each treatment, for each layer and total for each treatment as constructed.

Tons of Asphalt

Treatment	Layers 2	3	4	5	Total
174	256.67	252.39	184.48	179.67	873.21
190	150.00	147.50	107.81	105.00	510.31
196	210.00	206.50	184.48	179.67	780.65
202(control)	105.00	103.25	92.24	89.83	390.32

Table 5. Tons of binder in each treatment, for each layer and total for each treatment as constructed.

Tons of Binder

Treatment	Layers 2	3	4	5	Total
174	12.94	13.38	10.98	10.69	47.99
190	11.19	11.00	8.04	7.83	38.06
196	13.23	13.01	10.94	10.65	47.83
202(control)	5.95	5.85	4.65	4.53	20.98

Table 6. Tons of elemental sulphur in each treatment, for each layer and total for each treatment as constructed.

Tons of Sulphur

Treatment	2	Layers 3	4	5	Total
174	5.98	4.01	3.29	3.21	16.49
190	3.36	3.30	2.41	2.35	11.42
196	3.97	3.90	3.29	3.20	14.36
202(control)	0.00	0.00	0.00	0.00	00.00

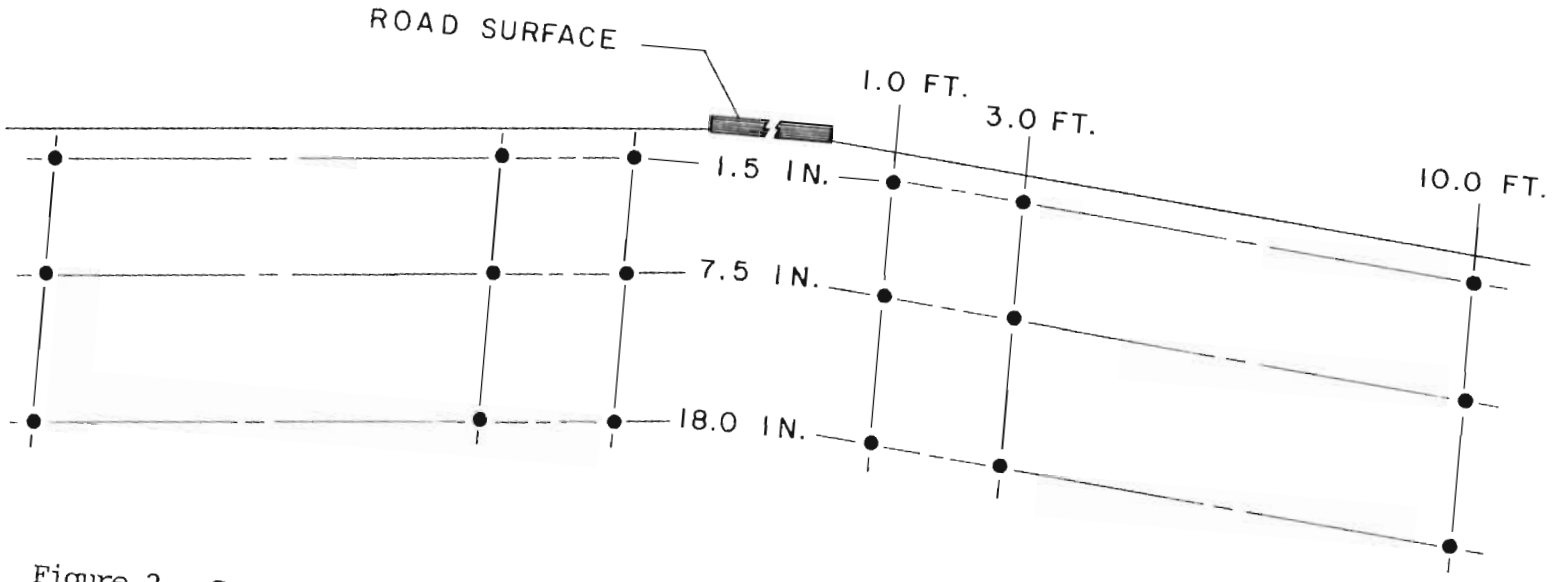

Figure 2. Cross section showing sampling points, distances from the road edge and the center of the sampling depths.

crew. The study areas were also sampled in May 1977, November 1977 and August 1978.

Laboratory Analyses

Exchangeable cations were extracted with ammonium acetate at pH 4.2 (currently used by the Texas Soil Testing Laboratory System) and measured by atomic absorption techniques.

Ammonium acetate at pH 4.2 was also used for sulfate extraction. Then the sulfate concentration was determined through turbidimetric measurement of a barium sulfate precipitate (Bardsley and Lancaster, 1965).

Available phosphorus was extracted using 0.002N sulfuric acid and determined colormetrically using the molybdate-blue method (Appendix C).

Soil reaction was measured with a pH meter using a 1:1 soil water suspension. The Bouyoucos method was used to determine soil texture (Appendix C).

Statistical Analysis

The data were subjected to a four-factor analysis of variance (ANOVA) with blocking on one of the factors, for each sampling period. The four factors used were treatment, side of highway, distance from the highway (ft.) and depth (in.) sampled. Side was blocked in all cases because it showed no significance when left unblocked. The Students Newman-Kuels (SNK) test ($p \geq .05$) was used to delineate where the differences were occuring.

To further determine which treatment or treatments may be en-

vironmentally sound, an ANOVA was performed on each treatment over the four sampling periods. The ANOVA accompanied by the SNK test ($p \geq .05$) revealed sulfate movement over the duration of the study. Finally, mean trends were observed for various soil variables to clarify any possible interrelationship caused by treatment effect over the duration of the study.

RESULTS AND DISCUSSIONS

The statistical analysis performed on the data collected from soil analysis revealed no significant sulfate concentration differences between the sides of the highway. Therefore, any variation due to side within the experiment was collapsed into the error term. The analyses of the samples collected during the study are found in Appendix B (Tables 1 to 8).

The primary analysis did reveal significant differences in sulfate concentration between treatments in all four sampling periods and between depths sampled within the soil in the May and November, 1977 sampling periods (Tables 1 to 4, Appendix A). To better estimate the cause of these differences, the secondary analysis focused on each treatment over the four sampling periods. The analysis revealed a significant difference in the soil sulfate concentration in treatments 190, 196 and the control. There was no significant difference in the soil sulfate concentration in treatment 174 (Tables 5 to 8, Appendix A).

After an analysis of variance, the SNK test was used to estimate when and where the soil sulfate concentration differences occurred, during the study (Tables 5 to 8, Appendix A).

The SNK analysis revealed no disparate patterns among treatments. However, if the mean soil sulfate concentrations for each treatment are compared over the duration of the study, two distinct trends emerge (Figure 3). Treatments 174 and 190 increased the soil sulfate

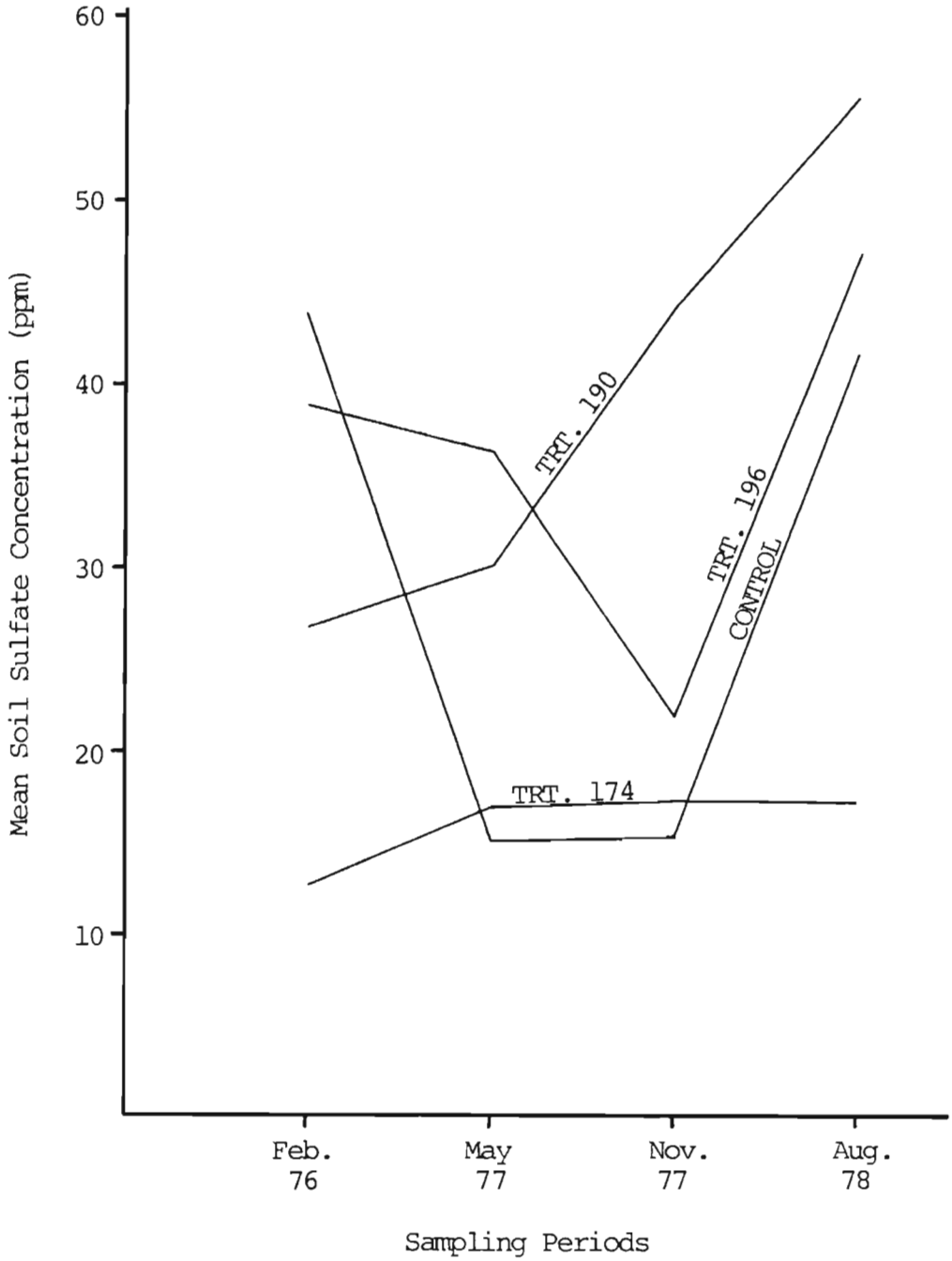

Figure 3. A graph showing the periodic fluctuation of mean sulfate concentration, of each treatment (TRT) and the control over the study duration.

concentration, whereas the soil sulfate concentration adjacent to the control location and treatment 196 fluctuated similarly throughout the study.

Table 7. Sulfate means for each treatment and sampling period.

Replications	202 (control)	174	190	196
		SO_4 ppm		
Feb.-76	44.056	12.667	26.833	38.772
May -77	15.056	16.778	30.056	36.444
Nov.-77	15.336	17.224	44.389	21.611
Aug.-78	41.556	17.113	55.612	47.056

These results suggest that the lower amount of sulfate during May 1977 and November 1977 in the control and treatment 196 could have been caused by the greater quantity of precipitation received prior to sampling. The average rainfall, calculated from the Alto, Lufkin and Nacogdoches records, show that monthly precipitation for the month of and two months prior to sampling were 42.4 and 26.9 percent greater in May 1977 and November 1977, respectively, than prior to sampling in February 1976 and August 1978 (U.S. Dept. Comm., NOAA, 1975, 1976, 1977 and 1978). During these periods of increased precipitation there was a significantly higher quantity of sulfate (35-38 ppm) in the 0-3" sampling depth as compared to the 3-12" and 12-24" sampling depths, where the sulfate concentration ranged from 16-21 ppm (Tables 2 and 3, Appendix A). Once the quantity of precipitation decreased, as it did prior to sampling in August 1978, the difference among depths became insignificant. The three distances (1 ft., 6 ft. and

10 ft.) sampled from the pavement showed no significant difference in soil sulfate concentration. One would expect more water to percolate into the soil closer to the pavement leaving a different concentration of sulfate at the one and six foot sampling distances than at the ten foot distance. Apparently, the grade away from the test section and the clayey nature of the soil allowed an even distribution of surface runoff over the distance sampled.

Treatment 196 was constructed identically to the control with two layers of the type "D" asphalt on top of two layers of hot sand, except that sulphur binding agent was substituted for the conventional binding agent. This treatment had fluctuations similar to the control, and the mean soil sulfate concentration increased by 8.3 ppm.

All four layers of treatment 174 consisted of the type "D" asphalt, raising the mean soil sulfate concentration by 4.6 ppm. This was an insignificant increase of soil sulfate over the duration of the study. The slow increase of sulfate associated with treatment 174 gives results similar to those achieved when fertilizing with gypsum or adding dolomite to increase soil pH. Therefore, use of the dual aggregate base material as in the type "D" asphalt may be beneficial to the sulfate deficient soil of the humid southeast.

The four layers of treatment 190 were constructed entirely with a hot sand single aggregate base material. This material significantly increased the quantity of sulfate within the soil. At the final sampling (August 1978), the sulfate concentration had increased by 107%. The high leaching rate exhibited by treatment 190 indicated

that the sand base material used in construction had little capacity to hold ions when exposed to weathering, although at sampling time the sulfate concentration showed no observable effect on the roadside ecosystems. Continued accumulation at the 107% rate over the life span of the road could result in soil acidification. The use of a calcium based single aggregate base material may alleviate the leaching problem by acting similar to a dolomitic lime. These results indicate that the base materials and permeability of the treatment may be influencing the leaching rate of the pavement.

Chemical analysis of the drainage ditch samples revealed a slight fluctuation in soil sulfate concentration. The lowest sulfate concentration occurred during the May 1977 and November 1977 sampling periods. This corresponds with the results obtained from the statistical analysis of the samples collected alongside the test section, again indicating that precipitation is one of the major factors influencing the eluviation of sulfates from the soil profile. Seventy percent of the samples collected in May 1977 were lower in sulfate than those taken in November 1977. Increased sulfate utilization by plants for growth and flowering and the quantity of precipitation during spring may have caused this phenomenon. Finally, the samples taken at the creek revealed a decrease in soil sulfate concentration. Therefore, it can be assumed that the sulfate leaching from the test section is not affecting the water quality of the creek or leaving the right of way.

CONCLUSIONS AND RECOMMENDATIONS

Summary

The mean quantity of sulfates in the soil beside treatment 196 (2 layers type "D" on top of 2 layers hot sand sulphur-asphalt) and the control (conventional asphalt) dropped in May 1977 and November 1977 during periods of increased precipitation. Treatment 174 (4 layers type "D" sulphur-asphalt) continued to increase the mean quantity of sulfate within the soil by 4.6 ppm. This increase was not a significant increase over the duration of the study. The pavement constructed with four layers of a single aggregate of hot sand sulphur-asphalt (treatment 190) significantly increased the mean soil sulfate concentration by 107% throughout the study.

There were no differences in the soil sulfate concentrations among the distances (1 ft., 6 ft. and 10 ft.) sampled. During May and November 1977 the 0-3 inch sampling depth had a significantly higher mean quantity of sulfates as compared to the 3-12 and 12-24 inch sampling depths.

Finally, samples taken from the drainage ditches and where all the roadside runoff leaves the right of way into a creek exhibited a decrease in soil sulfate concentration. This decrease in soil sulfate concentration indicates that the sulfate leaching from the test section had not left the right of way or entered the creek.

Recommendations

I recommend the use of sulphur-asphalt binder with the type "D"

base material throughout construction or the hot sand type "D" base materials alternated as was done in treatments 174 and 196, respectively. I also recommend caution be used when this binder is used with just the hot sand base material as in treatment 190, unless a calcareous material is mixed with the asphalt.

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APPENDIX A

RESULTS OF STATISTICAL ANALYSES

Table 1. ANOVA of sulfate data collected February 1976 and the Students Newman-Kuels multiple range test (SNK test).

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F
Treatment	3	10490.60	3496.87	8.43**
Side (Blocked) (S)	1	8.68	8.68	0.02
Distance (H)	2	440.03	220.01	0.53
Depth (D)	2	2150.36	1075.18	2.59
TxH	6	1209.53	201.55	0.49
TxD	6	5051.53	841.92	2.03
HxD	4	510.64	127.66	0.31
TxHxD	12	2440.47	203.37	0.49
Error	35	14513.82	414.68	
Total	71	36815.66		

**Significant at ($p \geq .01$)

SNK Test[1]

ppm SO_4	44.056	38.722	26.833	12.667
Treatment	202a	196a	190ab	174b

[1]Means not followed by the same letter are significantly different at the ($p \geq .05$) level.

Table 2. ANOVA of sulfate data collected May 1977 and the Students Newman-Kuels multiple range test (SNK test).

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F
Treatment (T)	3	5802.02	1934.02	7.27**
Side (Blocked) (S)	1	102.72	102.72	0.39
Distance (H)	2	175.75	87.88	0.33
Depth (D)	2	4413.25	2206.63	8.29**
TxH	6	1502.03	250.34	0.94
TxD	6	2323.53	387.25	1.46
HxD	4	560.00	140.00	0.53
TxHxD	12	3033.89	252.82	0.95
Error	35	9314.28	266.12	
Total	71	27227.47		

**Significant at ($p \geq .01$)

SNK Test[1]

ppm SO_4	15.056	30.056	36.444	16.778
Treatment	202b	196a	190a	174b
ppm SO_4	35.292	21.667	16.792	
depth	3"a	9"b	18"b	

[1]Means not followed by the same letter are significantly different at the ($p \geq .05$) level.

Table 3. ANOVA of sulfate data collected November 1977 and the Students Newman-Kuels multiple range test (SNK test).

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F
Treatment (T)	3	9733.57	3244.52	7.15**
Side (Blocked) (S)	1	786.72	786.72	1.73
Distance (H)	2	2061.79	1030.89	2.27
Depth (D)	2	7333.76	3666.88	8.08**
TxH	6	862.03	143.67	0.32
TxD	6	16.49	274.85	0.65
HxD	4	543.97	135.99	0.30
TxHxD	12	2092.34	174.36	0.38
Error	35	15891.38	454.04	
Total	71	40954.56		

**Significant at ($p \geq .01$)

SNK Test[1]

ppm SO_4	15.336	21.611	44.389	17.224
Treatment	202b	196b	190a	174b
ppm SO_4	38.875	18.417	16.627	
Treatment	3"a	6"b	18"b	

[1]Means not followed by the same letter are significantly different at the ($p \geq .05$) level.

Table 4. ANOVA of sulfate data collected August 1978 and the Students Newman-Kuels multiple range test (SNK Test).

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F
Treatment (T)	3	14747.47	4915.82	5.13*
Side (Blocked) (S)	1	.00	.00	0.00
Distance (H)	2	3670.54	1835.27	1.92
Depth (D)	2	1703.96	851.98	0.89
TxH	6	6466.01	1077.67	1.13
TxD	6	5125.58	854.26	0.89
HxD	4	826.82	206.71	0.22
TxHxD	12	2910.71	242.56	0.25
Error	35	33515.68	957.59	
Total	71	68966.77		

*Significant at ($p \geq .05$)

	SNK Test[1]			
ppm SO_4	41.556	47.056	55.612	17.113
Treatment	202a	196a	190a	174b

[1]Means not followed by the same letter are significantly different at the ($p \geq .05$) level.

Table 5. ANOVA of sulfate data collected in the four sampling periods for the control (202).

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F
Replication	3	13778.352	4592.7813	10.422**
Error	68	29966.766	440.688	
Total	71	43745.118		

**Significant at the ($p \geq .01$) level.

SNK Test[1]

ppm SO_4	44.056	15.056	15.336	41.556
sampling periods	Feb.76a	May77b	Nov.77b	Aug.78a

Table 6. ANOVA of sulfate data collected in the four sampling periods for treatment 196.

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F
Replication	3	6502.832	2167.6106	4.838**
Error	68	30465.770	448.0588	
Total	71	36968.602		

**Significant at the ($p \geq .01$) level.

SNK Test[1]

ppm SO_4	38.722	36.444	21.611	47.056
sampling periods	Feb.76a	May77ab	Nov.77b	Aug.78a

[1]Means not followed by the same letter are significantly different at ($p \geq .05$) level.

Table 7. ANOVA of sulfate data collected in the four sampling periods for treatment 190.

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F
Replication	3	8033.434	2677.811	3.112*
Error	68	58512.379	860.476	
Total	71	66545.813		

*Significant at the ($p \geq .05$) level.

	SNK Test[1]			
ppm SO_4	26.833	30.056	44.389	55.612
sampling periods	Feb.76b	May77b	Nov.77ab	Aug.78a

Table 8. ANOVA of sulfate data collected in the four sampling periods for treatment 174.

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F
Replication	3	259.222	86.641	0.744ns
Error	68	14245.949	209.499	
Total	71	14505.971		

ns - nonsignificant

[1]Means not followed by the same letter are significantly different at the ($p \geq .05$) level.

APPENDIX B

SULFATE AND pH ANALYTICAL RESULTS

Table 1. Analyses of roadside samples along Highway 69 Sulphur-Asphalt Binders Pavement Test, Lufkin, Texas (February 1976).

Sample Location*	SO_4 ppm	pH	Sample Location*	SO_4 ppm	pH
174-W-01-03	10	6.2	196-W-01-03	52	6.3
-09	10	7.0	-09	63	4.4
-18	4	4.0	-18	12	4.7
-06-03	16	4.1	-06-03	18	5.0
-09	16	3.9	-09	39	4.6
-18	10	4.0	-18	16	4.8
-10-03	8	3.6	-10-03	90	4.4
-09	14	4.2	-09	14	4.4
-18	23	3.2	-18	12	4.0
174-E-01-03	8	5.2	196-E-01-03	68	5.2
-09	16	3.9	-09	44	4.1
-18	19	3.6	-18	47	3.6
-06-03	11	4.0	-06-03	19	5.4
-09	11	4.0	-09	33	4.0
-18	14	4.0	-18	51	4.3
-10-03	8	5.4	-10-03	14	6.0
-09	14	3.9	-09	40	4.2
-18	16	4.2	-18	65	3.8
190-W-01-03	28	4.7	202-W-01-03	82	4.4
-09	27	4.9	-09	26	8.2
-18	22	4.8	-18	60	4.2
-06-03	23	4.2	-06-03	12	3.9
-09	20	4.4	-09	20	4.3
-18	19	4.7	-18	96	4.4
-10-03	28	6.0	-10-03	22	4.4
-09	22	4.3	-09	18	4.0
-18	44	4.4	-18	92	4.4
190-E-01-03	14	4.4	202-E-01-03	18	5.5
-09	18	4.6	-09	14	5.7
-18	42	4.8	-18	96	4.6
-06-03	10	6.7	-06-03	53	6.5
-09	30	4.8	-09	30	4.6
-18	36	4.8	-18	53	4.1
-10-03	29	7.8	-10-03	31	4.2
-09	26	4.8	-09	48	5.6
190-E-10-18	45	5.0	202-E-10-18	22	4.2

*Sample locations are as follows: 1st 3 numbers (174, 190, 196, 202) are highway stations; letter is West or East side of roadway; next 2 numbers (01, 06, 10) are the distances of the sample locations from the edge of the pavement; and the last 2 numbers (03, 09, 18) are the depths at which the samples were taken. For example, the first entry was taken from the 0 to 3 inch depth, 1 foot from the edge of the pavement on the west side of the highway at station 174.

Table 2. Analyses of samples taken in the drainage ditches adjacent to the Sulphur-Asphalt Binders Pavement Test, along Highway 69 near Lufkin, Texas in February 1976.

Sample Location*	SO_4 ppm	pH
174-W-D-03	26	4.6
-09	60	4.4
174-W-Slope	44	3.8
174-E-D-00	20	4.6
-03	5	4.6
188+50-W-out	58	4.8
190-E-D-03	31	4.6
-09	67	4.8
196-W-D-03	39	5.0
196-E-D-00	19	4.4
202-W-D-03	28	4.6
-Slope	36	4.6
202-E-D-03	66	4.6

*Sample location codes not explained in footnote to Table 1 are: D = ditch, Slope = backslope cut, -00 = a surface sample, out = drainage outlet off of right-of-way - all drainage from the test pavement is channeled to this point at 188+50.

Table 3. Analyses of roadside samples along Highway 69 Sulphur-Asphalt Binders Pavement Test, Lufkin, Texas (May 1977).

Sample Location*	SO_4 ppm	pH	Sample Location*	SO_4 ppm	pH
174-W-01-03	63	5.8	196-W-01-03	50	4.8
-09	45	4.0	-09	36	4.1
-18	14	3.7	-18	11	3.8
-06-03	15	4.9	-06-03	19	5.0
-09	33	3.6	-09	33	4.0
-18	8	3.6	-18	13	4.0
-10-03	28	5.0	-10-03	79	4.3
-09	6	3.9	-09	10	4.0
-18	7	3.8	-18	12	3.9
174-E-01-03	8	5.3	196-E-01-03	45	4.6
-09	8	4.6	-09	35	4.9
-18	10	3.6	-18	33	3.9
-06-03	4	5.1	-06-03	58	4.0
-09	14	3.9	-09	23	4.1
-18	10	3.2	-18	10	4.0
-10-03	8	6.1	-10-03	50	3.8
-09	9	3.2	-09	15	4.1
-18	12	3.6	-18	9	4.1
190-W-01-03	19	6.7	202-W-01-03	19	4.3
-09	22	4.1	-09	26	6.6
-18	23	4.1	-18	1	4.0
-06-03	47	4.3	-06-03	56	4.2
-09	26	4.4	-09	1	4.0
-18	15	4.2	-18	1	4.0
-10-03	30	5.1	-10-03	37	4.1
-09	22	5.1	-09	1	4.3
-18	13	4.1	-18	1	4.1
190-E-01-03	44	6.5	202-E-01-03	6	4.8
-09	22	4.0	-09	11	5.1
-18	55	4.2	-18	12	4.1
-06-03	54	5.0	-06-03	52	4.3
-09	34	4.2	-09	26	4.0
-18	62	4.3	-18	1	4.2
-10-03	55	4.1	-10-03	1	5.0
-09	50	4.2	-09	12	3.9
190-E-10-18	63	4.4	202-E-10-18	7	4.1

*Sample locations are as follows: 1st 3 numbers (174, 190, 196, 202) are highway stations; letter is West or East side of roadway; next 2 numbers (01, 06, 10) are the distances of the sample locations from the edge of the pavement; and the last 2 numbers (03, 09, 18) are the depths at which the samples were taken. For example, the first entry was taken from the 0 to 3 inch depth, 1 foot from the edge of the pavement on the west side of the highway at station 174.

Table 4. Analyses of samples taken in the drainage ditches adjacent to the Sulphur-Asphalt Binders Pavement Test, along Highway 69 near Lufkin, Texas in May 1977.

Sample Location*	SO_4 ppm	pH
174-W-D-03	13	3.9
-09	6	4.0
174-W-Slope	3	4.8
174-E-D-00	8	4.3
-03	7	4.3
188+50-W-out	58	4.2
190-E-D-03	23	4.7
-09	22	4.1
196-W-D-03	36	3.9
196-E-D-00	29	3.7
202-W-D-03	54	3.8
-Slope	14	4.6
202-E-D-03	1	5.0

*Sample location codes not explained in footnote to Table 1 are: D = ditch, Slope = backslope cut, -00 = a surface sample, out = drainage outlet off of right-of-way - all drainage from the test pavement is channeled to this point at 188+50.

Table 5. Analyses of roadside samples along Highway 69 Sulphur-Asphalt Binders Pavement Test, Lufkin, Texas (November 1977).

Sample Location*	SO_4 ppm	pH	Sample Location*	SO_4 ppm	pH
174-W-01-03	0	6.7	196-W-01-03	22	6.0
-09	16	3.9	-09	50	3.5
-18	1	3.6	-18	33	3.1
-06-03	44	5.2	-06-03	44	4.3
-09	49	3.5	-09	6	2.8
-18	1	3.4	-18	22	3.0
-10-03	22	3.3	-10-03	22	3.0
-09	0	3.6	-09	6	2.8
-18	6	3.6	-18	6	2.9
174-E-01-03	60	4.3	196-E-01-03	33	3.8
-09	22	3.5	-09	16	3.2
-18	39	3.4	-18	16	3.0
-06-03	11	5.1	-06-03	33	3.1
-09	11	3.6	-09	22	3.2
-18	0	3.5	-18	11	3.2
-10-03	11	3.6	-10-03	30	3.0
-09	1	3.5	-09	11	3.0
-18	16	3.5	-18	6	3.1
190-W-01-03	33	4.1	202-W-01-03	44	3.3
-09	33	3.0	-09	11	3.2
-18	11	4.1	-18	0	3.2
-06-03	60	3.4	-06-03	33	3.2
-09	22	3.0	-09	11	3.1
-18	22	3.0	-18	6	3.2
-10-03	22	3.1	-10-03	44	3.1
-09	22	3.4	-09	11	3.2
-18	33	3.5	-18	0	3.2
190-E-01-03	148	4.0	202-E-01-03	33	4.3
-09	50	3.6	-09	11	3.9
-18	66	3.7	-18	11	2.8
-06-03	44	3.4	-06-03	44	3.3
-09	33	3.6	-09	6	2.8
-18	33	3.2	-18	0	3.0
-10-03	85	3.5	-10-03	11	2.9
-09	22	3.7	-09	0	2.9
190-E-10-18	60	3.0	202-E-10-18	0	3.1

*Sample locations are as follows: 1st 3 numbers (174, 190, 196, 202) are highway stations; letter is West or East side of roadway; next 2 numbers (01, 06, 10) are the distances of the sample locations from the edge of the pavement; and the last 2 numbers (03, 09, 18) are the depths at which the samples were taken. For example, the first entry was taken from the 0 to 3 inch depth, 1 foot from the edge of the pavement on the west side of the highway at station 174.

Table 6. Analyses of samples taken in the drainage ditches adjacent to the Sulphur-Asphalt Binders Pavement Test, along Highway 69 near Lufkin, Texas in November 1977.

Sample Location*	SO_4 ppm	pH
174-W-D-03	26	4.6
-09	11	3.1
174-W-Slope	11	2.8
174-E-D-00	11	3.2
-03	6	3.2
188+50-W-out	11	5.0
190-E-D-03	33	3.5
-09	25	3.3
196-W-D-03	42	2.9
196-E-D-00	0	4.7
202-W-D-03	42	3.2
-Slope	33	3.2
202-E-D-03	22	2.9

*Sample location codes not explained in footnote to Table 1 are: D = ditch, Slope = backslope cut, -00 = a surface sample, out = drainage outlet off of right-of-way - all drainage from the test pavement is channeled to this point at 188+50.

Table 7. Analyses of roadside samples along Highway 69 Sulphur-Asphalt Binders Pavement Test, Lufkin, Texas (August 1978).

Sample Location*	SO_4 ppm	pH	Sample Location	SO_4 ppm	pH
174-W-01-03	22	6.7	196-W-01-03	55	4.1
-09	22	4.2	-09	44	3.6
-18	11	3.5	-18	11	3.6
-06-03	44	4.0	-06-03	66	4.1
-09	33	3.8	-09	55	3.5
-18	11	3.9	-18	33	3.4
-10-03	55	4.3	-10-03	77	3.8
-09	22	3.9	-09	22	3.6
-18	11	3.9	-18	33	3.6
174-E-01-03	1	5.1	196-E-01-03	77	4.9
-09	11	5.2	-09	99	4.6
-18	1	4.4	-18	44	3.6
-06-03	1	4.7	-06-03	22	4.0
-09	11	4.7	-09	77	3.4
-18	22	3.9	-18	11	3.3
-10-03	11	4.6	-10-03	22	3.7
-09	11	3.9	-09	66	3.1
-18	11	3.9	-18	33	3.3
190-W-01-03	77	4.6	202-W-01-03	77	3.3
-09	55	4.0	-09	55	5.8
-18	22	3.8	-18	33	4.0
-06-03	55	3.8	-06-03	33	3.9
-09	44	3.7	-09	33	3.4
-18	33	3.7	-18	22	3.7
-10-03	55	3.8	-10-03	66	3.5
-09	33	3.8	-09	44	3.3
-18	33	3.5	-18	55	3.4
190-E-01-03	33	6.1	202-E-01-03	55	4.1
-09	132	6.3	-09	44	4.3
-18	198	4.9	-18	33	4.2
-06-03	22	6.1	-06-03	22	4.4
-09	55	5.9	-09	66	3.9
-18	55	4.5	-18	33	3.7
-10-03	1	4.8	-10-03	22	4.5
-09	44	4.8	-09	33	3.7
190-E-10-18	55	4.2	202-E-10-18	22	3.7

*Sample locations are as follows: 1st 3 numbers (174, 190, 196, 202) are highway stations; letter is West or East side of roadway; next 2 numbers (01, 06, 10) are the distances of the sample locations from the edge of the pavement; and the last 2 numbers (03, 09, 18) are the depths at which the samples were taken. For example, the first entry was taken from the 0 to 3 inch depth, 1 foot from the edge of the pavement on the west side of the highway at station 174.

Table 8. Analyses of samples taken in the drainage ditches adjacent to the Sulphur-Asphalt Binders Pavement Test, along Highway 69 near Lufkin, Texas in August 1978.

Sample Location*	SO_4 ppm	pH
174-W-D-03	33	3.6
-09	22	3.6
174-W-Slope	44	3.4
174-E-D-00	33	3.5
-03	33	3.6
188+50-W-out	22	5.3
190-E-D-03	44	3.9
-09	33	3.6
196-W-D-03	88	3.6
196-E-D-00	1	6.1
202-W-D-03	11	5.2
-Slope	11	4.4
202-E-D-03	33	3.7

*Sample location codes not explained in footnote to Table 1 are: D = ditch, Slope = backslope cut, -00 = a surface sample, out = drainage outlet off of right-of-way - all drainage from the test pavement is channeled to this point at 188+50.

APPENDIX C

METHODS OF SOIL ANALYSES

ACETATE-SOLUBLE SULFATE[1]

Reagents

1. Extracting solution - 1.4 mol. ammonium acetate, 1.0 mol. hydrochloric acid, pH 4.2: To five liters of distilled water add 1494 ml glacial acetic acid and mix. While stirring, add 1750 ml concentrated ammonium hydroxide and allow to cool. Add 1460 ml concentrated hydrochloric acid and allow to cool. Bring to a volume of 18 liters with distilled water and stir. Adjust to pH 4.2.

2. Norit "A" activated charcoal: Wash the charcoal with extracting solution until it is free of sulfate.

3. Acid "seed" solution: 6 N HCl containing 20 ppm of sulphur as K_2SO_4.

4. Barium chloride, 20 to 60 mesh crystals.

5. Potassium sulfate, reagent grade.

Standards

1. Stock solution: Dissolve 0.5434 g reagent grade K_2SO_4 in extracting solution in a 1-liter volumetric flask, and bring to 1 liter with extracting solution. The resulting solution contains 100 ppm of S.

2. Standard solutions: By using different proportions of extracting solution and the 100 ppm stock solution prepare standards containing 0, 2, 5, 10, 20, 25, 30, and 40 ppm S.

Procedure

1. Shake 10 g of 20 mesh soil with 25 ml of extracting solution in a 250 ml Erlenmeyer flask for 30 minutes.

2. Add 0.25 g of charcoal and shake for an additional 3 minutes.

3. Filter the soil suspension.

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[1]Derived from Bardsley and Lancaster, 1965.

4. Pipette 10 ml of filtrate into a test tube.

5. Add 1 ml of "seed" solution, swirl.

6. Add 0.5 g of barium chloride crystals. Let stand for 1 minute, then swirl.

7. Read light transmission on a colorimeter or spectrophotometer at a wavelength of 420 mu within 2 to 8 minutes and calculate the sulfate concentration using a standard curve.

8. Prepare the standard curve by running the standard solutions through steps 2 - 7 and plotting the percent transmission for each standard on graph paper.

DETERMINATION OF AVAILABLE PHOSPHORUS

Reagents

1. Extracting solution: Prepare a stock solution of 0.1 N H_2SO_4 by titrating against standard alkali. Dilute convenienent volume to 0.002 N (1 ml H_2SO_4 per 18 liters distilled water 0.002 N) and buffer with 3 g $(NH_4)_2SO_4$ per liter (54 g).

2. Sulfomolybdic Acid: Dissolve 25 g ammonium molybdate in 200 ml distilled water at 60°c. Dilute 275 ml concentrated H_2SO_4 to 800 ml. When cool, mix both solutions and cool again. Dilute mixture to 1000 ml.

3. Stannous chloride solution: Dissolve 25 g $SnCl_2$ in 100 ml concentrated HCl. Dilute to 1000 ml. Store in brown bottle and protect from air with a layer of mineral oil.

Standards

1. Stock solution: Dissolve 0.2195 g NH_4HPO_4 and dilute to 1000 ml with extracting solution. This contains 50 ppm P. Dilute 50 ml ppm P to 500 ml. This final solution contains 5 ppm.

2. Standard solutions: By using different proportions of extracting solution and the 5 ppm P stock solution, prepare standards containing 0, 0.2, 0.4, 0.6, 0.8 and 1.6 ppm P.

Procedure

1. Place 1.0 g soil in a 250 ml Erlenmeyer flask.

2. Add 100 ml of 0.002 N H_2SO_4.

3. Shake 1/2 hour and filter. Do not rinse or add any more solution.

4. Place 50 ml aliquot into a 250 ml Erlenmeyer flask.

5. Add 2 ml of sulfomolybdic acid.

6. Add 3 drops of $SnCl_2$.

7. Pour some of the colored solution into a test tube or colorimeter tube.

8. Determine intensity of color on colorimeter at 660 mu. The concentration of P in the solution is determined from a standard curve made up with the standard solutions containing known amounts of P.

TEXTURE ANALYSIS (BOUYOUCOS METHOD)

Reagent

1. Sodium metaphosphate - saturated solution.

Procedure

1. Weigh 50 g (oven dry) soil (100 g of coarse textured soil). Add to mixing cup and fill 1/2 with water. Add 20 ml of sodium metaphosphate. The Na replaces the cations on the surface of the clay and promotes an increase in the net negative charge, causing the particles to disperse by repelling each other.

2. Mix on stirrer until soft aggregates are broken down (10 - 15 minutes). This enables the soil fraction to become separated and free in suspension.

3. Transfer to Bouyoucos cylinder and fill to lower mark (upper mark if 100 g are used). Keep hydrometer in solution while filling.

4. Remove hydrometer, place stopper in top of cylinder, and shake cylinder. Place cylinder on bench and record time. At 20 seconds insert hydrometer and take reading at 40 seconds. Sand size fraction (larger than 0.05 mm) settles out in to seconds. Silt and clay fraction remain in suspension.

5. Remove hydrometer and record temperature. For each degree above that on the hydrometer (67° or 68°F) add 0.2 to the reading, subtract 0.2 for each degree less than is listed.

6. Calculate percent sand: Wt. of sample - corrected hydrometer reading = wt. of sand. Wt. of sand/wt. of sample x 100 = percent sand. The hydrometer is calibrated to read in grams of soil particles in suspension. Thus, the 40 second reading gives the grams of silt and clay in suspension.

7. Repeat hydrometer reading, temperature reading and correction at 2 hours to give the weight of the clay fraction remaining in suspension.

8. Calculate percent clay: Percent clay = corr. hyd. reading/ wt. of sample x 100.

9. Calculate percent silt: 100 - (percent sand + percent clay) = percent silt.

10. Determine class name or texture from textural triangle.

SULFATE LEACHING FROM SULPHUR BINDING AGENTS

IN EXPERIMENTAL ASPHALT PAVEMENT,

ANGELINA COUNTY, TEXAS

APPROVED:

Dr. Kenneth G. Watterston,
Thesis Director

Dr. James E. Howard,
Committee Member

Dr. David L. Kulhavy,
Committee Member

Glen T. Clayton, Dean of the
Graduate School

SULFATE LEACHING FROM SULPHUR BINDING AGENTS IN EXPERIMENTAL ASPHALT PAVEMENT, ANGELINA COUNTY, TEXAS

by

PETER LYNN BLANCHETTE, B.S.F.

Presented to the Faculty of the Graduate School of
Stephen F. Austin State University
In Partial Fulfillment
of the Requirements

For the Degree of
Master of Science in Forestry

STEPHEN F. AUSTIN STATE UNIVERSITY
May 1985

ABSTRACT

Sulfate leaching from sulphur-binding agents used in asphalt pavement was significantly higher ($p \geq .05$) from four layers of a mixture of a single aggregate base material of hot sand, when compared to all other treatments. Analysis of the data showed that the other treatments had no overall effect on the soil sulfates. Four layers of a single aggregate of hot sand increased the quantity of sulfates in the soil by 107%.

I recommend the use of sulphur-asphalt binder with the type "D" base material throughout construction or when two layers of the type "D" base material are placed on top of two layers of hot sand. I also recommend caution be used when this binder is used with just the hot sand base material, unless a calcareous material is mixed with the asphalt.

VITA

Peter Lynn Blanchette was born in Springfield, Massachusetts, on April 9, 1954, the son of Penny P. Blanchette and Dr. Joseph Alfred Blanchette. After completing his work at Cushing Academy, Ashburnham, Massachusetts, in 1972, he entered Stephen F. Austin State University in January 1973. He received a Bachelors of Science in Forestry from Stephen F. Austin State University in December 1976. In January 1977, he entered Graduate School at Stephen F. Austin State University.

Permanent Address: 8714 Charing Cross Lane
Dallas, Texas 75238

This thesis was typed by Barbara K. Blanchette.